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Laboratory Research

Evaluation of Resin Bond Strength to Yttria-stabilized Tetragonal Zirconia and Framework Marginal Fit: Comparison of Different Surface Conditionings

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Clinical Relevance

The low-fusing glass application followed by hydrofluoric acid etching and silanization seems to be a promising method for improving the resin bond strength to yttria-stabilized tetragonal zirconia, but the adhesion to this substrate is still a challenge.

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SUMMARY

The purpose of this study was to evaluate the effect of different surface treatments of yttria-stabilized tetragonal zirconia (Y-TZP) on bond strength durability and marginal discrepancies. For adhesion testing, 144 specimens of VITA In-Ceram YZ ceramic for InLab were obtained (5.25×3.75×4.5 mm) and divided into six groups (n=24) according to the surface treatment: 1) Control (CRTL): untreated; 2) SIL: tribochemical silica coating (CoJet system, 3M/ESPE AG); 3) V1+HF: spray application of low-fusing porcelain glaze (V1, VITA Akzent Spray Glaze) followed by etching with hydrofluoric acid (HF) (one minute); 4) V1+SIL: V1 glazing (VITA Akzent Spray Glaze) followed

by tribochemical silica coating; 5) V2+HF: brush application of low-fusing porcelain glaze (VITA Akzent Glaze) plus etching with HF (one minute); and 6) V2+SIL: V2 glazing (VITA Akzent Glaze) plus tribochemical silica coating. After all treatments, the surfaces were silanized for five minutes (ESPE-SIL) and cementation was performed using Panavia F (Kuraray). Half of the specimens in each treatment were tested 24 hours after cementation (dry), with the other half subjected to storage (150 days) and thermocycling (12,000×) (aging), and then a shear test was carried out (1 mm/min). The micromorphological (digital optical profilometry and scanning electron microscopy) and elemental analyses of the treated surfaces were performed. The inner surfaces of 60 Y-TZP infrastructures were conditioned and marginal fit was evaluated. The statistical analysis revealed that the groups treated via surface glaze application followed by hydrofluoric acid etching and silanization showed the highest bond strength (in dry and aging conditions), but the bond strengths were affected by aging. The highest marginal discrepancies were observed in the groups receiving glaze (117.4 ± 29.6 to 105.8 ± 12.2 μm) when compared to other groups (55.3 ± 8.7 and 55 ± 8.5 μm). Low-fusing porcelain glaze + hydrofluoric acid etching changed the morphology of the Y-TZP ceramic and improved the adhesion to the resin cement, but obtaining high and stable bond values to Y-TZP remains challenging. Marginal discrepancies increased with glazing.

INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) has been increasingly used as an infrastructure for metal-free prostheses as a result of its microstructure, which provides excellent mechanical properties, such as high flexural strength, fracture toughness, and phase transformation toughening (through the stresses generated by cracks).[1] These frameworks are suitable for fixed partial dentures with up to two anterior and posterior pontics.[2]

In the case of adhesive cementation of Y-TZP–based restorations, the surface conditioning is a challenge because these materials are highly crystalline (free of the glassy phase),[3,4] unlike the feldspathic ceramics (rich in silicon oxides), which have more established bonding mechanisms.[4,5] A successful bond between the feldspathic ceramic and resin cement is obtained through the formation of chemical bonds and micromechanical retention between both materials.[6] This bonding is well established for ceramics with a high content of silica by selective conditioning of the glassy phase when the ceramics are exposed to hydrofluoric acid, which increases the surface area and surface roughness, improves the wettability and surface free energy, and exposes a greater amount of silicon oxides.[5] The application of coupling agents based on methacryloxypropyltrimethoxysilane produces increased wettability and forms siloxane bonds between the silica content of the ceramic and the organic matrix of the resin cement.[7,8] As a result of the absence of the glassy phase in the Y-TZP ceramics, etching with hydrofluoric acid does not generate sufficient surface changes and does not promote micromechanical retention, nor does it make the surface chemically reactive.[3,4]

To overcome this problem, surface pretreatment for Y-TZP ceramics has been suggested.[9-15] Airborne particle abrasion with aluminum oxide or tribochemical silica coating associated with the use of silanes and 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-containing resin cements were evaluated and the results were found to be contradictory. Passos and others,[16] Nishigawa and others,[17] and May and others[18] found better bond strength results when the surface was silica-coated and silanized. Matinlinna and others[19] found low and unstable bond strength results with the use of silanization as the surface treatment. Currently, another questionable and conflicting aspect of bonding these substrates concerns the strong impact that air-particle abrasion may cause on the surface of Y-TZP. Some studies[20-22] observed detrimental effects of air abrasion on the strength of Y-TZP ceramics, depending on the development of microcracks and possible premature catastrophic fracture of the restorations. However, other studies[23-27] have reported increased mechanical strengths of these ceramics after airborne particle abrasion.

The use of adhesion promoters (primers) with experimental zirconia has been considered as an alternative protocol to promote this bonding, although these primers still have adhesive strength results[12,19] that are less than those obtained with the cementation of feldspathic ceramics.[4,28] Another treatment that has been tried is surface modification of ceramic Y-TZP via vitrification, which renders it rich in silicon oxides. This film could be selectively conditioned or totally removed to make the surface retentive and chemically reactive, similar to the bonding mechanisms of feldspathic ceramics.[9-11,14,15,29-33] Promising results of Y-TZP/resin cement bond strengths have been observed after the ceramic surface has been glazed and subjected to air-particle abrasion with aluminum oxide and silanization[11,14,15] or etching with hydrofluoric acid.[10,15,31-33] However, a controversial issue still requires an answer: how durable is the bonding promoted by these innovative methods? In addition to this question, there is still no recent evidence regarding the influence of the application of a silicon oxide–based film on the intaglio surface on the marginal fit of Y-TZP crowns.

According to Denissem and others[34] and Kokubo and others,[35] the clinical success of restorations depends on many factors, including the internal and marginal fit of the ceramic crown. Sharp marginal discrepancies between the restoration and prepared tooth interfere with the longevity of the restorative treatment.

Cement that is exposed to the oral environment serves as a weak point between the restoration and the prepared tooth.[36] The marginal fit of Y-TZP crowns was evaluated by Att and others,[37] who found marginal gaps of between 64 and 78 μm. A cervical misfit of up to 119 μm is considered clinically acceptable,[38-42] which makes it important to observe the effect caused by the vitrification of crowns on the marginal fit of Y-TZP crowns.

Within this context, this present study proposed a "new" technique for the treatment of the cementation surface for Y-TZP ceramics (glass film application) and compared that new method to methods routinely used for ceramic conditioning. The marginal fit of an infrastructure made of Y-TZP ceramics under different conditionings was also evaluated to examine whether this technique could be clinically viable. The hypotheses were the following: glazing of the Y-TZP crown intaglio surface would improve adhesion of the ceramic to resin cement; storage/thermal cycling would degrade the bond strengths; and the glazing would not interfere with the marginal fit.

MATERIALS AND METHODS

Production of Specimens

Y-TZP ceramic blocks (14×15×20 mm; VITA In-Ceram 2000 YZ cubes for InLab) were prepared using a cutting machine (ISOMET 1000, Buehler Ltd, Lake Bluff, IL, USA), resulting in 144 blocks (7×6×5 mm). These blocks were sintered (ZYrcomat,-VITA Zahnfabrik Oven, Bad Sackingen, Germany) according to the manufacturer's guidelines, obtaining specimens with standardized final dimensions (5.25×3.75×4.5 mm).

The samples were polished with wet sandpaper of decreasing granulation (400 and 600 grit up to 1200 grit). All of the specimens were submitted to an ultrasonic bath (Vitasonic, VITA Zahnfabrik, Bad Sackingen, Germany) for five minutes in distilled water and were then randomly divided into six groups according to the surface treatments. The specimens were then embedded in chemically activated acrylic resin for the shear bond strength test, keeping the surface for adhesion exposed. All of the specimens were subjected to an ultrasonic bath again for five minutes in distilled water and then cleaned with alcohol.

Y-TZP Surface Treatments

The 144 Y-TZP samples were allocated into six groups, according to the method of conditioning of the bonding surface (n=24), as follows: 1) Control group (CRTL): no surface treatment (control group). 2) SIL: silica-coating by air-borne particle abrasion with silica-coated aluminum oxide particles (CoJet®-Sand, 3M ESPE AG, Seefeld, Germany), using a microetcher device (Cojet-Prep™, 3M ESPE AG). The distance between the ceramic surface and the device point was standardized at 10 mm and had an inclination of 45°, with the aid of a device (pressure 2.5 bar for 10 seconds). 3) V1+HF: low-fusing porcelain glaze (Glaze Spray VITA Akzent, VITA Zahnfabrik, Bad Sackingen, Germany): glaze spray was applied twice and sintered according to the manufacturer's guidelines. Subsequently, the glazed surface was etched with 9% hydrofluoric acid gel (HF) for one minute (Ultradent Porcelain Etch, South Jordan, Ultradent), rinsed with air-water spray, and dried. Finally, the samples were cleaned in a sonic bath (five minutes in distilled H_2O). 4) V1+SIL: The Y-TZP surface was glazed as described for the V1+HF group (Glaze Spray VITA Akzent, VITA Zahnfabrik). Then the surface was conditioned with a tribochemical silica coating method (as described for the SIL group). 5) V2+HF: low-fusing porcelain glaze (Glaze VITA Akzent, VITA Zahnfabrik). According to the manufacturer's instructions, the glaze was prepared and applied once on the surface using a brush, followed by sintering, as recommended. The glazed surface was etched with 9% HF for one minute, rinsed with air-water spray, and dried. Finally, the samples were cleaned in a sonic bath (five minutes in distilled H_2O). 6) V2+SIL: The surface was glazed as described for the V2+HF group (Glaze VITA Akzent, VITA

Table 1: *Testing Groups for Bond Strength Evaluation, Considering the Two Studied Factors (Y-TZP Surface Conditioning [in Six Levels] and Storage Condition [in Two Levels])*

Groups	Y-TZP Surface Conditionings (n=12)	Aging	Bond Results[a]	Student *t*-Test[b]	Surface Roughness[c]
Control (CTRL)	Without surface conditioning	Baseline (no aging)	2.3 ± 1.2 D		0.14
SIL	Tribochemical silica coating		7.9 ± 2.6 C	0.0001	0.35
V1+HF	Glazing 1 + etching with 9% hydrofluoric acid		13.3 ± 4.1 AB	0.019	3.37
V1+SIL	Glazing 1 + silica coating		9.2 ± 1.9 BC	0.0001	0.41
V2+HF	Glazing 2 + etching with 9% hydrofluoric acid		17.8 ± 5.5 A	0.030	3.79
V2+SIL	Glazing 2 + silica coating		12.5 ± 4.3 BC	0.123, ns	0.40
CTRL(TC)	Without surface conditioning	Aging	0.01 ± 0.0 c		
SIL(TC)	Tribochemical silica coating		2.2 ± 1.8 bc		
V1+HF(TC)	Glazing 1 + etching with 9% hydrofluoric acid		9.3 ± 2.7 a		
V1+SIL(TC)	Glazing 1 + silica coating		4.7 ± 1.2 b		
V2+HF(TC)	Glazing 2 + etching with 9% hydrofluoric acid		12.5 ± 4.2 a		
V2+SIL(TC)	Glazing 2 + silica coating		9.8 ± 2.8 a		

Abbreviations: CTRL, control group; SIL, tribochemical silica coating; TC, during aging; V1+HF, spray application of low-fusing porcelain glaze followed by etching with hydrofluoric acid (HF); V1+SIL, V1 glazing followed by tribochemical silica coating; V2+HF, brush application of low-fusing porcelain glaze plus etching with HF; V2+SIL, V2 glazing plus SIL; Y-TZP, Yttria-stabilized tetragonal ceramic.

[a] *Means (±standard deviation[SD]) of the bond strength data (MPa) and Tukey test are presented. Uppercase letters indicate statistically similar Baseline groups. Lowercase letters indicate statistically similar Aging groups.*

[b] p *values for comparison (Student t-test) between the groups submitted to the same Y-TZP surface conditioning under different storage conditions (*p-value<0.05 *represents the means of bond strength have significant difference; ns, no difference).*

[c] *The surface roughness results (*R_a*, in μm).*

Zahnfabrik) and conditioned with a tribochemical silica coating method (as described for the SIL group).

After conditioning, all of the specimens were submitted to silanization for five minutes (ESPE-SIL silane, 3M/ESPE).

Cement Application

After conditioning of the Y-TZP bonding surface, a cement was mixed (Panavia F 2.0, Kuraray Medical Inc, Okayama, Japan) and applied using a syringe (Centrix Syringe system, Dentsply Detrey, Konstanz, Germany) inside a metal matrix (diameter: 3.5 mm; height: 2 mm) that had been placed on the ceramic surface. The resin cement was light-activated for 40 seconds (XL 3000, 3M/ESPE; light intensity=600 mW/cm^2) and the matrix was removed.

Aging (Storage and Thermal Cycling)

Half of the specimens in each treatment were tested 24 hours after cementation (dry condition) and the other half were subjected to storage (150 days) and thermocycling (12,000 cycles; 5°C and 55°C water baths; 30 seconds each bath; two seconds of transition) (aging condition); then the shear bond strength testing was performed. Twelve groups were formed (six Y-TZP surface conditionings and two aging conditions) (Table 1).

Shear Bond Strength Test

The samples were subjected to a shear bond strength test in a universal testing machine (EMIC DL-1000, São José dos Pinhais, Brazil). A knife-shaped indenter applied the load at a cross-head speed of 1 mm/min. A metal frame was used for holding each specimen to guarantee that the adhesive interface was parallel to the path of the knife and as near as possible to the long axis of the knife. The bond strength was calculated according to the formula $\sigma = F/A$, in which σ represents the strength (MPa), F is the load for the specimen failure (N), and A is the specimen interfacial area (mm^2). The bonded area (A) was uniform at 9.42 mm^2 ($A=\pi \times r^2$, in which π=3.14 and r=radius of the bonded area [1.5 mm]).

Failure Analysis

All specimens that were submitted to the shear testing were observed under 50×-200× magnifications using a stereomicroscope (Discovery V20, Zeiss, Gottingen, Germany) to observe their failure pattern. Specimens with representative fractures were chosen for microscopic analysis. The chosen specimens were mounted on metallic stubs, sputtercoated with gold (Denton Vacuum, DESK II), and observed under a scanning electron microscope (SEM; JEOL JSM-6360, Tokyo, Japan) at different magnifications.

The failures were classified as follows: 1) Adhesive failure: failure at the ceramic-cement interface; 2) Cohesive failure in the cement; 3) Cohesive failure in the ceramic; and 4) Mixed failure: adhesive failure combined with cohesive failure in the cement.

Surface Characterization

For the qualitative analysis of the surface after treatment, two samples (301.3×229.2 μm) from each surface treatment were evaluated under a SEM (JEOL JSM-6360, Tokyo, Japan) (1000× magnification) and a digital optical profilometer (Wyko, Model NT 1100, Veeco, Tucson, USA). The profilometer was connected to a PC containing imaging software (Vision 32, Veeco, USA) for data analysis of R_a roughness (arithmetic mean of all peaks and valleys found during reading of the sample). The measurements of the roughness parameters were performed at a magnification of 20× on five representative areas of each sample.

Chemical Analysis

Constituents and trace elements on the ceramic surfaces of each group were determined using energy dispersive x-ray spectroscopy (EDS; JEOL JSM-6360), which was performed on two areas per sample in each group. The concentration of elements was calculated based on the mass concentration of the elements present in each reading.

Marginal Fit Analysis

A metal die prepared for a full crown was used. Impressions were taken using addition silicone (Elite HD Putty Soft Normal Setting and Light Body Normal Setting, Zhermack SpA, Badia Polesine, Italy) through the double impression technique, following the recommendations of the manufacturer. The models were poured with type IV die stone for the Cerec system (No. 10300206, CAM-base®, Denton AG, Deutschland, Germany). Using the Cerec 3D program, 60 ceramic infrastructures were fabricated from 60 prefabricated ceramic blocks (VITA In-Ceram YZ for InLab, VITA Zanhfabrik).

The analysis of marginal discrepancy (vertical distance from one point of the crown margin to a point on the preparation margin) was evaluated using an optical microscope (Mitutoyo IM, 176-581A) with a magnification of 250× at 50 points along the margins of the infrastructure.

Statistical Analysis

Statistical software was used (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL, USA) for data analysis.

Bond strength data from dry and aging conditions separately were submitted to one-way analysis of variance (ANOVA) and Tukey test (α=0.05). In addition, the groups were compared 2-2 to elucidate the isolated effect of storage for each surface treatment, using the Student *t*-test ($p<0.05$).

To compare the six experimental conditions on the outcome "marginal discrepancy," the data were submitted to Kruskal-Wallis and Dunn multiple comparison tests (5%).

RESULTS

Descriptive statistics (means and standard deviations) of the bond strength data and the Tukey test ($p<0.05$) are shown in Table 1 and Figure 1.

With regard to the pretest failures, Table 2 indicates that all of the specimens from the control group failed during the aging procedures.

In both the dry and aging conditions separately, the one-way ANOVA of bond strength data showed the surface conditionings (p=0.00001) had a significant influence.

Tukey analysis showed that the surface treatment Glazing 2 + HF (V2+HF) presented the highest bond strengths and the control group the lowest bond strengths in both the dry and aging conditions.

The pairwise comparison (Student *t*-test; Table 1) shows that the bond strengths from all of the surface conditionings decreased after aging, except in the case of V2+SIL.

The measurements of roughness parameters (arithmetic mean of all peaks and valleys) encountered during the reading of the samples are described in Table 1. There was a greater roughness in the groups with silica deposition (vitrification), followed by those with etching. The micrographs confirm the micromorphological changes (Figure 2E,F). When glass application and acid etching were observed, significant pits were formed by selective etching of the glass film, which seemed to promote better micromechanical retention of the resin cement. In the groups with silanization (Figure 2C,D,G,H), a slight increase in roughness occurred compared to the control (Figure 2A,B).

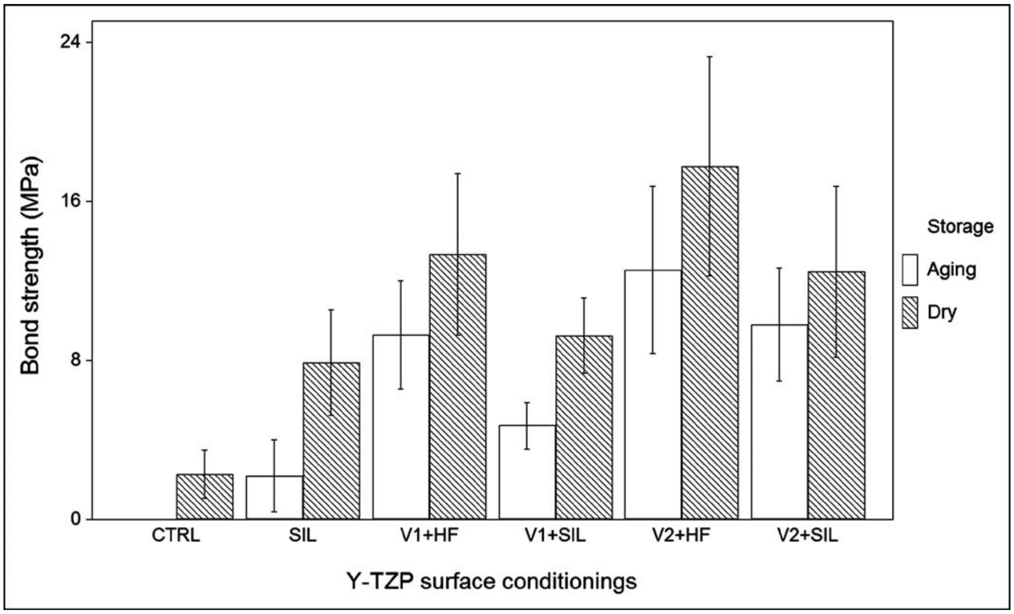

Figure 1. *Mean shear bond strength (MPa) and standard deviation (bars) of bond strength data for the different Y-TZP surface conditionings and storage conditions.*

The EDS elemental chemical analysis (Table 3) indicated a stronger presence of silica in the groups with glass application.

The failure analyses (Table 4) indicated that mixed failure was the predominant failure type. However, the CTRL and CTRL(aging) groups presented 75% and 100% adhesive failures, respectively. Representative failures are presented in Figure 3.

Table 2: *Number (N) and Percentages (%) of the Specimens (sp), Which Spontaneously Prematurely Failed (FPT) During Aging (TC), and Total N of the Specimens Submitted to Bond Strength Test (SBS)*

Groups	N of sp	N (%) of Pretest Failure During Aging	Total N (%) of sp Tested
CTRL	12	0 (0)	12 (100)
SIL	12	0 (0)	12 (100)
V1+HF	12	0 (0)	12 (100)
V1+SIL	12	0 (0)	12 (100)
V2+HF	12	0 (0)	12 (100)
V2+SIL	12	0 (0)	12 (100)
CTRL(TC)	12	12 (100)	0 (0)
SIL(TC)	12	3 (25)	9 (75)
V1+HF(TC)	12	0 (0)	12 (100)
V1+SIL(TC)	12	0 (0)	12 (100)
V2+HF(TC)	12	0 (0)	12 (100)
V2+SIL(TC)	12	0 (0)	12 (100)

Abbreviations: CTRL, control group; SIL, tribochemical silica coating; V1+HF, spray application of low-fusing porcelain glaze followed by etching with hydrofluoric acid (HF); V1+SIL, V1 glazing followed by tribochemical silica coating; V2+HF, brush application of low-fusing porcelain glaze plus etching with HF; V2+SIL, V2 glazing plus SIL.

The medians of the marginal misfit data are presented in Table 5. The Kruskal-Wallis test showed the surface conditionings ($p=0.001$) had significant influence on the results. The Dunn test multiple comparisons showed that glazing groups had the highest values of marginal misfit when compared to other groups.

A preliminary analysis of the different thicknesses and surface glazes was performed (VITA Aktent spray glaze and VITA Akzent glaze) after the application on the Y-TZP ceramic surface, as shown in Figures 4 and 5. The thickness of the glaze was approximately 10 μm.

DISCUSSION

In studies of bond strength, a relevant aspect concerns the influence of premature failure of specimens on the primary outcome (bond strength). It has been reported[43] that the analysis of variance of bond strength data becomes more consistent when the specimens lost prematurely are considered for statistical analysis and that their omission can provide a significant overestimation of the results. Many studies in the literature do not consider premature failures; consequently, those findings might not accurately reflect the bonding performance,[44,45] leading to false interpretations. Thus, there is a need to report the percentage of premature

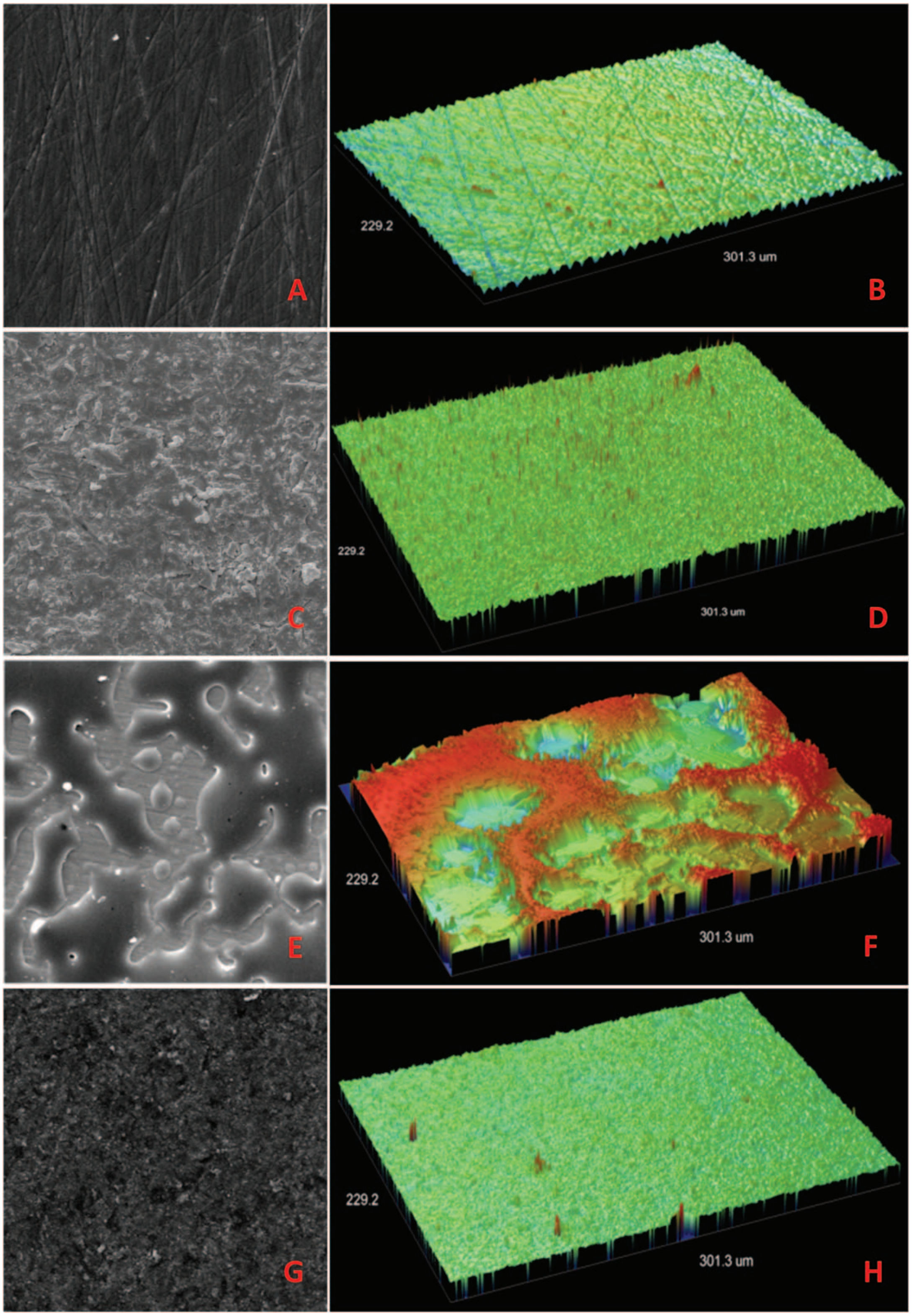

Figure 2. *Representative images of Y-TZP surface. Surface without conditioning (control group) under SEM (1000×) (A) and digital optical profilometer (B). Surface after silica coating (SIL group) under SEM (1000×) (C) and digital optical profilometer (D). Surface after glazing followed by etching with 9% hydrofluoric acid under SEM (1000×) (E) and digital optical profilometer (F) (representative of two glaze approaches). Surface after glazing followed by silica coating under SEM (1000×) (G) and digital optical profilometer (H) (representative of two glaze approaches).*

Table 3: *Elemental Chemical Analysis (%) of the Y-TZP Surface Submitted to the Different Conditionings (Energy Dispersive X-ray Spectroscopy [EDS] Analysis)*

	CTRL	SIL	V1+HF	V1+SIL	V2+HF	V2+SIL
Zr	71.54	55.28	52.35	51.04	49.62	21.50
O	27.07	32.76	33.22	32.73	33.03	40.86
Si	—	8.74	11.15	11.57	12.09	25.04
K	—	—	1.13	2.36	2.56	5.34
Ca	—	—	—	1.79	2.18	5.61
Al	—	2.15	2.15	0.51	0.51	1.63
Y	1.39	1.07	—	—	—	—

Abbreviations: Al, aluminum; Ca, calcium; CTRL, control group; K, potassium; O, oxygen; Si, silicon; SIL, tribochemical silica coating; V1+HF, spray application of low-fusing porcelain glaze followed by etching with hydrofluoric acid (HF); V1+SIL, V1 glazing followed by tribochemical silica coating; V2+HF, brush application of low-fusing porcelain glaze plus etching with HF; V2+SIL, V2 glazing plus SIL; Y, yttrium; Zr, zirconium.

specimen failures due to their weak bonding, even before testing.[46] According to Table 2, the percentage of specimens lost during the aging procedures reached 100% for the control group. This dramatic percentage of pretest failures is related to the low values of bond strength in this group, detected during testing of the "dry" specimens (2.3 MPa). The group that received silanization [V2+SIL(aging)] had 25% spontaneous failures during aging, which explains the low values observed after aging.

In order to consider the deleterious effects of specimens with pretest failures, the value of 0.01 MPa was arbitrarily assigned to each specimen "lost," and these specimens were considered in the statistical analysis. This value was considered only to allow for statistical calculations. These "losses" indicate greater susceptibility to degradation of the interface, and assigning an arbitrary value allows a more precise interpretation of the adhesive performance between the substrate and adhesive, preventing misinterpretations.[45]

The study of bond strength of Y-TZP restorations has been the subject of several works,[9,12,17-19,33,47-49] as the difficulty of bonding to the adhesive cement is reported as one of the main limitations.[3,9,33] For this reason, several studies have been developed to improve the bond strength of Y-TZP ceramics to the adhesive cement using different surface conditionings, such as modifying the surface through air-abrasion with particles of alumina or silica (tribosilanization)[16-18] zirconia primers,[12,19] glazing,[10,15] or deposition of silica films.[9,11,14,29,30-33,50] Even though the manufacturers allow the use of conventional cements (zinc phosphate and glass ionomer) for the cementation of Y-TZP crowns,[51] it is known that adhesive cementation provides greater retention from microretentions provided by the surface conditioning of dental hard tissues and restorative

Table 4: *Number and Percentage (%) of Specimens (sp) Submitted to the Shear Test and of Type of Failure*

Groups	sp Tested	Type of Failure			
		Adhesive	Mixed	Cohesive (Cement)	Cohesive (Ceramic)
CTRL	12 (100)	9 (75)	3 (25)	0 (0)	0 (0)
SIL	12 (100)	2 (16.6)	10 (83.4)	0 (0)	0 (0)
V1+HF	12 (100)	0 (0)	11 (91.6)	1 (8.4)	0 (0)
V1+SIL	12 (100)	0 (0)	12 (100)	0 (0)	0 (0)
V2+HF	12 (100)	0 (0)	11 (91.6)	1 (8.4)	0 (0)
V2+SIL	12 (100)	0 (0)	12 (100)	0 (0)	0 (0)
CTRL(TC)	0 (0)	12 (100)	0 (0)	0 (0)	0 (0)
SIL(TC)	9 (75)	11 (91.6)	1 (8.4)	0 (0)	0 (0)
V1+HF(TC)	12 (100)	0 (0)	12 (100)	0 (0)	0 (0)
V1+SIL(TC)	12 (100)	3 (25)	9 (75)	0 (0)	0 (0)
V2+HF(TC)	12 (100)	0 (0)	12 (100)	0 (0)	0 (0)
V2+SIL(TC)	12 (100)	1 (8.4)	11 (91.6)	0 (0)	0 (0)

Abbreviations: CTRL, control group; SIL, tribochemical silica coating; TC, during aging; V1+HF, spray application of low-fusing porcelain glaze followed by etching with hydrofluoric acid (HF); V1+SIL, V1 glazing followed by tribochemical silica coating; V2+HF, brush application of low-fusing porcelain glaze plus etching with HF; V2+SIL, V2 glazing plus SIL.

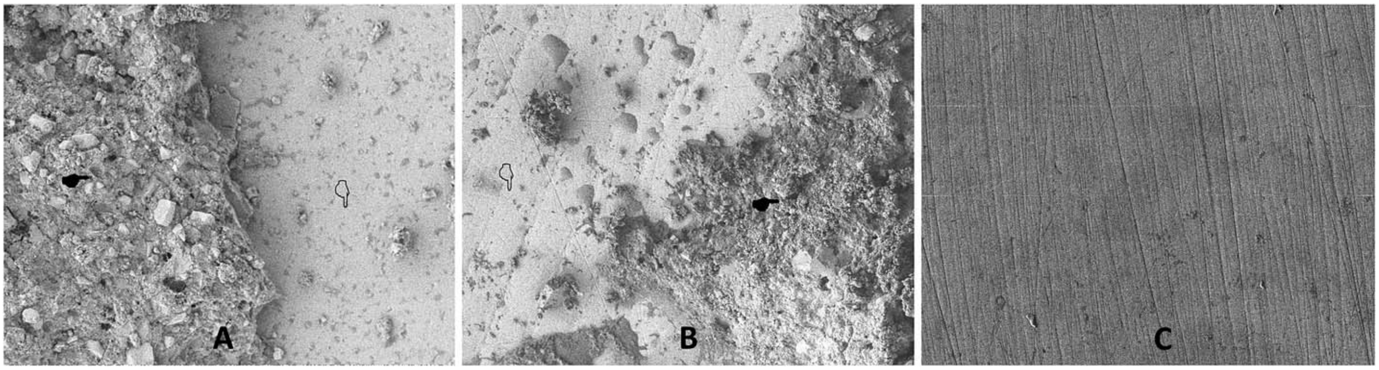

Figure 3. *Representative micrographs of fractured surface from tested specimens (400× magnification): (A, B) mixed failure: ☚ indicates the resin cement and ☟ means Y-TZP surface. (C) Adhesive failure: Y-TZP surface cement free.*

materials,[52,53] while also providing lower solubility of cement and lower marginal leakage,[54] which is the main culprit in the replacement of restorations.[55] Given these characteristics, this current study modified the surface of Y-TZP in order to improve the bond strength to resin cements.

When considering the approaches presented in the literature, the application of low-fusing glass followed by hydrofluoric acid etching appears to be a promising method when conditioning Y-TZP.[9-11,14,15,29-33,50] After this treatment, the Y-TZP behaves as a silicon oxide–based ceramic from the standpoint of the adhesive, in that the treated surface presents of the possibility of micromechanical bonding by acid etching while providing a chemically reactive surface via the chemical bonds of silane, which serves as a link between the deposited/applied silica and the organic matrix of resin cements (siloxane bond).[4-6,19,29,56,57] Abou-shelib and others[29] coated the surface of Y-TZP with low-fusion glass (30% silicon, 13% titanium, 8% aluminum, 3% potassium, 1% rubidium, 1% magnesium, and the remnants of O_2) and found increased bond strengths when compared to the control group (without glass application). Valentino and others[15] found higher bond strength values when the Y-TZP surface was glass-coated and acid-conditioned. Kitayama and others[11] modified the surface of zirconia, covering it with a thin layer of glass ceramics (100 μm), and achieved better bond strength results to resin cement by acquiring the characteristics of a feldspathic ceramic.[4-6]

In this current study, two strategies for surface glazing from the same manufacturer were used (two types of commercially available glazing agents) with similar chemical compositions, but with different modes of application: spray (Spray Glaze, VITA Akzent: Glazing 1) and powder + liquid (liquid-powder Glaze: VITA Akzent: Glazing 2).

The glazing surface treatment followed by hydrofluoric acid etching and silanization improved the bond strength when compared to the control group (Table 1). Even though the application method of glass application suffered a significant reduction in bond strength after aging, the values obtained in the aged condition were higher when compared to those of other groups (Table 1).

These results can be explained by two mechanisms: 1) bonding via micromechanical retention: the strong micromorphological changes (increased roughness) optimized the interaction between the adhesive and substrate[4,28,58-61] (Figure 2E,F), or 2) chemical bond: the increased percentage of silica on the surface (Table 3) may have contributed to an enhanced physical and chemical interaction between the glassy film–silane-resin cement.[2,4,19,29,57]

However, according to Student t test, the bond strengths from all surface treatments were reduced significantly after long-term aging, except for the treatment of V2 + silanization (Table 1). These present findings demonstrate the strong difficulty

Table 5: *Medians of Marginal Fit Data of the Y-TZP Frameworks After Different Conditionings of the Intaglio Surface and Dunn Test[a] (α=5%)*

Y-TZP Surface Conditionings	Medians[a]
Glazing 1 + silica coating (V1+SIL)	106.15 A
Glazing 1 + acid etching (V1+HF)	103.90 A
Glazing 2 + silica coating (V2+SIL)	105.20 A
Glazing 2 + acid etching (V2+HF)	103.20 A
Control group	56.0 B
Silica coating (SIL)	54.5 B

[a] *Dunn test. Same letters = no significant difference. Different letters = significant difference.*

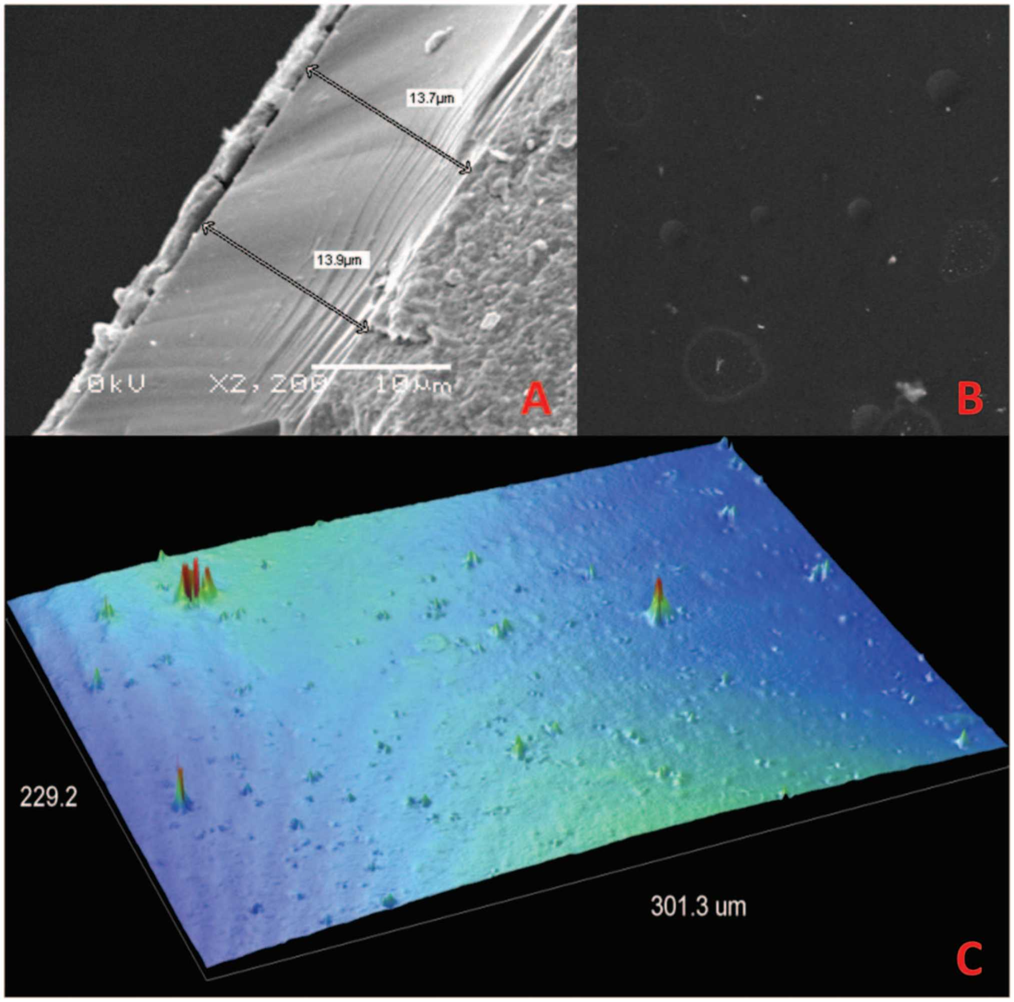

Figure 4. *Representative images of Y-TZP surface after glazing obtained from SEM (1000×) at transversal view (A) and surface view (B) and from digital optical profilometer (C).*

associated with stabilizing resin adhesion to the Y-TZP–based ceramic. Aboushelib and others[29,30] found no influence of storage on the values of bond strength after one, two, three, and four weeks of storage when performing vitrification of the Y-TZP ceramics and cementing with an MDP-containing resin cement (Panavia F), which may have occurred because of the short period for degradation of the

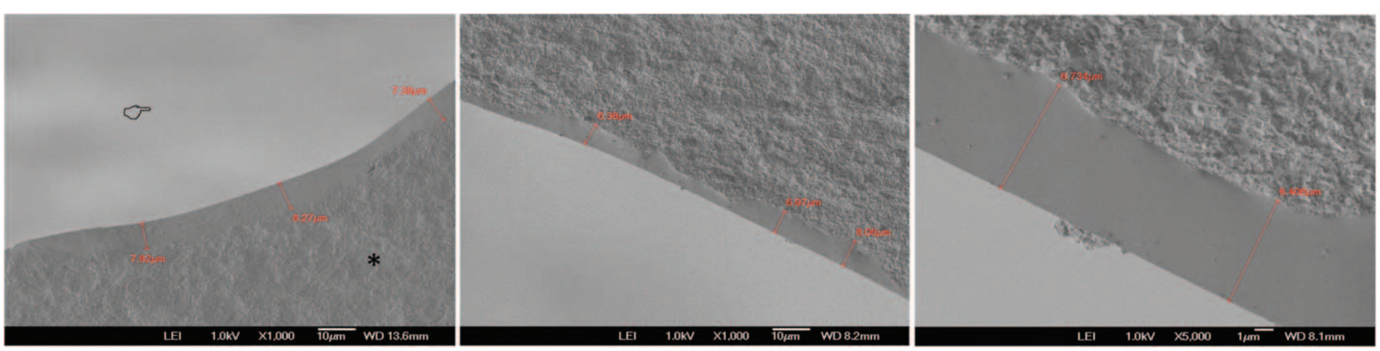

Figure 5. *Representative micrographs of the transversal surface of Y-TZP frameworks submitted to glazing ("spray" approach). The frameworks were fractured in order for it to be possible to produce these images: Whiter zone (☞) represents the Y-TZP material and blacker zone (*) indicates the glaze material.*

interface cement/Y-TZP. Another study[31] with a storage time of 90 days found a decrease in bond strength. Despite the reduction in bond strength values after storage/thermocycling, the bond strengths for the groups that received surface treatments were higher than the bond strengths of the control group, which showed 100% spontaneous failures and a high percentage of adhesive failures (dry and aging conditions), demonstrating the weak adhesive interaction of the resin cement to the Y-TZP surface (Table 4; Figure 5).

In the groups with glass coating associated with the air-abrasion of silica particles (SIL group), the deposition of silica particles can be observed, associated with a uniform surface roughness (R_a, 0.41-0.40) (Figure 2C,D). The current authors hypothesized that the "soft" glass surface may optimize the incrustation of silica particles onto the glass-coated Y-TZP surface; however, the results were lower than those for the groups submitted to etching with hydrofluoric acid. Thus, the possibility of introducing cracks in the ceramic through the impact pressure of the air-borne particle abrasion seems to be an unnecessary risk because the bond strength was higher when conditioning with hydrofluoric acid.[20-22]

Taking into account that the marginal discrepancy evaluation is influenced, among other factors, by the thickness of the cement[62] and that the technique of cementation affects the marginal fit,[63] cementation was performed in the current study to assess marginal fit. Moreover, it has been shown that the cement layer usually covers the points of measurement, making it difficult to perform accurate measurements.[64-66]

The control and tribosilicatization (SIL) groups showed statistically lower marginal discrepancies when compared to the groups that received glass application. A plausible explanation for this may be that the glass applied on the intaglio surface of the Y-TZP infrastructure formed a layer thick enough to interfere with seating of the infrastructure. This reflects the difficulty in standardizing the glaze application inside the Y-TZP infrastructure. This fact is a major limitation of this present study. However, despite this limitation, the mean values of marginal fit obtained in the current study appear to be within the range of clinically acceptable marginal discrepancies.[38-41,67] Besides, taking into account the marginal misfit results of control and tribosilicatization (SIL) groups, it can be hypothesized that the nano-film deposition approach of Y-TZP silicon oxide surface conditioning likely has no impact on marginal misfit.

Finally, given the present results, it appears that the glass application on the surface of Y-TZP, followed by hydrofluoric acid etching, is promising with regard to bond improvement. However, some issues should be further studied to better establish a plausible technique: the bond durability, the effect on the marginal discrepancies, the influence of conditioning on the mechanical behavior of the material, the duration of etching with hydrofluoric acid, and the technique of glass application on the intaglio surface of the restorations. Clinical studies may be needed to confirm the clinical feasibility of these procedures.

CONCLUSION

- The application of low-fusing glass on the surface of Y-TZP, followed by hydrofluoric acid etching and silanization, significantly improved the bond between Y-TZP and resin cement, but the resin adhesion was unstable.
- Storage/thermocycling affected the bond strength results, demonstrating bond instability to Y-TZP.
- The glass application increased the marginal discrepancies when compared to the results associated with other treatments.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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